

## METHOD OF PRODUCING ALUMINUM NITRIDE SINTERED BODY

## 2. Claim

1. A method of producing an aluminum nitride sintered body, comprising:

a step of forming into a required shape a raw material powder prepared by adding at least 0.1 to 3.0% by weight of amorphous carbon to an aluminum nitride powder, and

a step of subjecting the obtained compact to deoxidation treatment in a non-oxidizing atmosphere at a temperature of 1,500°C to 1,700°C and subsequently sintering the body at a temperature of 1,700°C to 2,200°C.

## 3. Detailed Description of the Invention

[Purpose of the Invention]

(Field of the Invention)

The present invention relates to a method of producing an aluminum nitride sintered body excellent in thermal conductivity.

(Prior Art)

Ceramics sintered bodies having aluminum nitride as the primary component have high thermal conductivities that are 5 to 10 times that of an aluminum oxide sintered body, and exhibit various excellent properties such as excellent heat-radiating properties, high electrical insulating properties, low

dielectric constants, as well as excellent corrosion resistance and thermal shock resistance properties, and thus they have received attention as materials for various electrical and electric parts and structural materials.

In particular, because a ceramics sintered body is excellent in heat-radiating properties as well as being close in coefficient of thermal expansion to a silicon single crystal, it is expected as the insulating substrate for semiconductor devices in place of an alumina sintered body of insufficient heat-radiating properties and beryllia sintered body, which requires complicated handling due to toxicity.

Incidentally, such an aluminum nitride sintered body is usually manufactured by adding an appropriate amount of a sintering aid, that is compound of an alkali earth metal or a rare earth metal, further adding to this an organic binder to fabricate a compact of a required shape, subjecting this compact to degreasing treatment, and subsequently sintering the body in a non-oxidizing atmosphere near a normal atmospheric pressure.

#### [Problems to be Solved by the Invention]

A demand for aluminum nitride described above as semiconductor substrates is now increasing year by year, and cost-reduction measures are desired.

Therefore, the possibility of the cost reduction is being studied in all respects including aluminum nitride raw materials, sintering aids, and treatment processes.

First possible considerations include use of low-cost aluminum nitride raw materials and exclusion of addition of comparatively expensive sintering aids.

However, the firing without addition of a sintering aid causes oxygen to remain in the aluminum nitride sintered body, which lowers thermal conductivity and denseness.

Also, because the optimization of high thermal conductivity of aluminum nitride is required in addition to cost reduction, high-purity aluminum nitride with a less content of impurities such as oxygen needs to be used as the raw material in order to obtain high thermal conductivity without addition of a sintering aid.

However, a raw material of high purity is, as a matter of course, expensive, which prevents the attainment of the purpose of cost reduction.

Thus, attaining simultaneously cost reduction and improvement of thermal conductivity becomes a task.

The present invention has been made to accomplish this task, and the object thereof is to provide a method of producing an aluminum nitride sintered body that offers an aluminum nitride sintered body without depending on the presence or absence of a sintering aid or the purity of an aluminum nitride raw material.

[Constitution of the Invention]

(Means for Solving the Problems)

A method of producing an aluminum nitride sintered body

of the present invention comprises a step of forming into a required shape a raw material powder prepared by adding at least 0.1 to 3.0% by weight of amorphous carbon to an aluminum nitride powder, and a step of subjecting the obtained compact to deoxidation treatment in a non-oxidizing atmosphere at a temperature of 1,500°C to 1,700°C and subsequently sintering the body at a temperature of 1,700°C to 2,200°C.

In the present invention, a raw material aluminum nitride to be used is not particularly limited, and an aluminum nitride sintered body of high thermal conductivity can be obtained even without use of a sintering aid.

In addition, a sintering aid may be used together with amorphous carbon of the present invention.

The amount of amorphous carbon to be added to an aluminum nitride powder is preferably 0.1 to 3.0% by weight, more preferably 0.5 to 1.5% by weight, relative to the amount of the aluminum nitride powder.

The reason why amorphous carbon is used is that crystalline carbon has a high melting point and thus is not dissipated in the degreasing and firing steps and remains in the sintered body, i.e. it is difficult to remove it.

Rendering the amount of such amorphous carbon to be added to be 0.1% by weight or less cannot obtain a body of high thermal conductivity, while making the amount exceed 3.0% by weight causes amorphous carbon to remain in the sintered body and

consequently to reduce the attributes, and thus the addition is preferably made in the above-mentioned range.

Then, the amount of amorphous carbon to be added is appropriately adjusted according to the oxygen content of an aluminum nitride raw material powder to be used.

Usable amorphous carbon sources include charcoal, animal charcoal, and coke, and also include materials that become amorphous carbon by heating such as a phenol resin.

A compact of a required shape is fabricated by adding, as required, an organic binder to this raw material powder and using a usual molding method such as the doctor blade method, press molding method, casting molding method, injection molding method or extrusion method.

Subsequently, the compact thus obtained is degreased and then is subjected to deoxidation treatment in a non-oxidizing atmosphere at a temperature of 1,500°C to 1,700°C.

Non-oxidizing atmospheres include those of nitrogen and argon, and the treatment is conducted by keeping at a temperature of 1,500°C to 1,700°C for about 0.5 to 2 hours.

After deoxidation treatment, the body is further sintered in a non-oxidizing atmosphere described above at a temperature of 1,700°C to 2,200°C for about 1 to 48 hours.

This can provide an aluminum nitride sintered body with a thermal conductivity of about 100 W/m·k or more.

(Operation)

The thermal conductivity of an aluminum nitride sintered body depends on the oxygen content. The presence of many oxygen atoms on the surfaces of or inside the aluminum nitride powders drops the thermal conductivity due to phonon scattering.

Thus, it is effective to remove oxygen in the aluminum nitride sintered body as much as possible to improve its thermal conductivity. For this reason, the present invention involves the addition of amorphous carbon to the aluminum nitride sintered body.

This amorphous carbon combines with oxygen in the aluminum nitride particles by means of heating to generate carbon monoxide or carbon dioxide and dissipate.

This enables the reduction of the oxygen content in an aluminum nitride sintered body, leading to the production of an aluminum nitride sintered body of high thermal conductivity, even in the case of no addition of a sintering aid or the use of an aluminum nitride raw material powder of low purity.

#### [Examples]

Examples of the present invention will be described hereinafter.

##### Example 1

To an aluminum nitride powder containing 0.8% by weight of oxygen was added 0.5% by weight of carbon black (amorphous) relative to the amount of the aluminum nitride powder.

Further to this raw material powder were added suitable amounts of organic binder and dispersing agent, and the resulting material was kneaded, and then a sheet-like compact was fabricated by the doctor blade method.

This compact was subjected to degreasing treatment in air at 550°C for two hours.

Thereafter, this degreased body was subjected to deoxidation treatment by heating in a nitrogen atmosphere at 1,600°C for 45 minutes.

Then, it was fired in a nitrogen atmosphere at 2,000°C for 6 hours to fabricate an aluminum nitride sintered body.

The aluminum nitride sintered body produced in this manner was found to have 130 to 150 W/m·k of thermal conductivity by the laser flash method and to have 35.5 kgf/mm<sup>2</sup> of three-point bending strength at ambient temperature.

In addition, due to no addition of a sintering aid, the liquid phase did not seep and the surface roughness Ra of the aluminum nitride sintered body was as very good as 0.3 μm.

#### Example 2

To an aluminum nitride powder containing 1.0% by weight of oxygen was added 0.55% by weight of carbon black relative to the amount of the aluminum nitride powder.

Further to this raw material powder were added suitable amounts of organic binder and dispersing agent, and the resulting material was kneaded, and then a sheet-like compact was

fabricated by the doctor blade method.

This compact was subjected to degreasing treatment in air at 550°C for two hours.

Thereafter, this degreased body was subjected to deoxidation treatment by heating in a nitrogen atmosphere at 1,600°C for 45 minutes.

Then, it was fired in a nitrogen atmosphere at a pressure of 5 atm at 2,000°C for 6 hours to fabricate an aluminum nitride sintered body.

The aluminum nitride sintered body produced in this manner was found, under the same conditions as in Example 1, to have 130 to 145 W/m·k of thermal conductivity and to have 34.8 kgf/mm<sup>2</sup> of three-point bending strength at ambient temperature.

In addition, the surface roughness Ra of the aluminum nitride sintered body was 0.3 μm.

### Example 3

To an aluminum nitride powder containing 1.3% by weight of oxygen was added 1.0% by weight of carbon black relative to the amount of the aluminum nitride powder.

Further to this raw material powder were added suitable amounts of sintering aid, organic binder and dispersing agent, and the resulting material was kneaded, and then a sheet-like compact was fabricated by the doctor blade method.

This compact was subjected to degreasing treatment in air at 550°C for two hours.



Thereafter, this degreased body was subjected to deoxidation treatment by heating in a nitrogen atmosphere at 1,600°C for 45 minutes.

Then, it was fired in a nitrogen atmosphere at a pressure of 5 atm at 2,000°C for 6 hours to fabricate an aluminum nitride sintered body.

The aluminum nitride sintered body produced in this manner was found, under the same conditions as in Example 1, to have 120 to 140 W/m·k of thermal conductivity and to have 36 kgf/mm<sup>2</sup> of three-point bending strength at ambient temperature.

Incidentally, in this Example, a liquid component seeping to the surface of the aluminum nitride sintered body due to the addition of a sintering aid was observed, and the surface roughness Ra of the aluminum nitride sintered body was worsened to 0.4 as compared with the cases where a sintering aid was not added. However, the denseness was excellent.

These results show that addition of amorphous carbon to an aluminum nitride powder enabled the removal of oxygen in an aluminum nitride sintered body and the fabrication of an aluminum nitride sintered body of high thermal conductivity regardless of the presence or absence of a sintering aid and the purity of the aluminum nitride raw material powder.

Furthermore, firing with the addition of amorphous carbon only and without use of a sintering aid enabled the obtainment of an aluminum nitride sintered body of even surface accuracy

without liquid phase seepage, and it also contributed to the improvement of workability.

[Advantages of the Invention]

As described thus far, a method of producing an aluminum nitride sintered body of the present invention enables the obtainment of an aluminum nitride sintered body of high thermal conductivity even if an expensive sintering aid or a high-purity raw material powder is not utilized.

Thus, it is capable of attaining cost reduction with the excellent quality maintained.